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# ACOUSTIC EMISSION DURING CHROMIUM PLATING

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The authors used acoustic emission to study crack formation during the electrodeposition of chromium over a range of temperatures and current densities which characterize the transition from high contraction (HC) to low contraction (LC) chromium. Several characteristics of the acoustic emissions were examined, including the amplitude distribution, signal energy, emission rate, count rate, and frequency spectrum. The acoustic emission technique (CONT'D ON REVERSE)		

## 20. ABSTRACT (CONT'D)

detected the onset of cracking and yielded useful information regarding the number of cracks. Such information acquired during the actual deposition process can be an invaluable aid in determining the quality of the deposit and in developing improved chromium coatings.

Experimental data were acquired and analyzed using commercial acoustic emission instrumentation, including standard piezoelectric transducers. The deposition bath was a standard aqueous solution of 250 ~~g/l~~<sup>g/l</sup> chromic acid and 2.5 g/l sulfuric acid. The solution temperature and deposition current density were varied to obtain deposits of differing crack content. The substrate was electropolished carbon steel.

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## INTRODUCTION

The use of acoustic emission to study crack formation and propagation has been well documented in numerous applications. However, very little work has been done to date using acoustic emission to study crack formation during the electrodeposition of chromium. This study represents an effort to develop the use of acoustic emission as a tool to monitor the condition of the chromium deposit during the actual deposition process.

Beattie defines acoustic emission as acoustic waves generated by a material when subjected to an external stimulus (ref 1). The acoustic wave is not generated by an external source and then introduced into the material, as is done in the field of ultrasonics. Rather, some mechanism in the material itself generates the acoustic wave in response to an external stimulus. Two typical external stimuli are a high stress level and a change in temperature. A material's response to these stimuli often involves a rapid, collective motion of atoms which generates an acoustic wave. Those mechanisms which generate acoustic waves include fracture of crystallites, crack formation and growth, motion of dislocations, fracture of inclusions, phase transformations, boiling, and electrical discharges (ref 1).

In the case of chromium electrodeposition, the external stimulus is the build-up of high internal tensile stress in the deposit during the deposition process. Although the reasons for this build-up of stresses are not fully understood, it is well known that they are relieved by the formation of cracks. It is this crack formation which generates acoustic emissions that may be readily observed.

<sup>1</sup>A. G. Beattie, "Acoustic Emission, Principles and Instrumentation," Journal of Acoustic Emission, Vol. 2, April 1983, pp. 95-128.

Of the many signal parameters to monitor, the most useful are usually the emission count rate, cumulative number of counts, and the amplitude. The emission count is the number of times the signal exceeds a predetermined threshold. A single emission event is usually counted several times with some weighting in favor of events of larger energy (ref 2). Amplitude distributions have also proven useful in interpreting acoustic emission activity (refs 3-5).

#### EXPERIMENTAL PROCEDURE

In both high contraction (HC) and low contraction (LC) cases, chromium was deposited galvanostatically in a standard plating solution of 250 g/l  $\text{CrO}_3$  and 2.5 g/l  $\text{H}_2\text{SO}_4$ . The anode was a sheet of platinum. The chromium coatings were deposited on a flat plate of 4340 steel 7.6 cm by 1.9 cm by 0.16 cm with an exposed circle of area 2.6  $\text{cm}^2$ . The plate had been polished with fine silicon carbide paper and the exposed area electropolished. HC chromium was deposited using a cathodic current of 30  $\text{A}/\text{dm}^2$  in a solution held at 55°C. LC chromium was deposited using a cathodic current of 120  $\text{A}/\text{dm}^2$  in a solution held at 85°C. A coating of intermediate crack density was deposited using a cathodic current of 45  $\text{A}/\text{dm}^2$  in a solution held at 70°C.

The acoustic emission was observed using standard commercial equipment (Figure 1) consisting of a resonant transducer (150 kHz, sensitivity of -70 dB re 1 V/ $\mu\text{Bar}$ , Physical Acoustics Corp. model R15); a preamplifier with a band-

<sup>2</sup>B. J. Brindley, J. Holt, and I. G. Palmer, "Acoustic Emission III - The Use of Ring-Down Counting," Non-Destructive Testing, Vol. 6, No. 6, December 1973.

<sup>3</sup>Y. Nakamura, C. L. Veach, and B. O. McCauley, "Amplitude Distribution of Acoustic Emission Signals," ASTM STP 505, 1972, pp. 164-186.

<sup>4</sup>J. Holt, D. J. Goddard, and I. G. Palmer, "Methods of Measurement and Assessment of the Acoustic Emission Activity From the Deformation of Low Alloy Steels," NDT International, Vol. 14, No. 2, April 1981, pp. 49-58.

<sup>5</sup>A. A. Pollock, "Acoustic Emission Amplitude Distributions," International Advances in Nondestructive Testing, Vol. 7, 1981, pp. 215-239.

pass filter (100-300 kHz, gain of 40 dB, Physical Acoustics Corp. model 1220A); amplifier and analyzer (Physical Acoustics Corp. model 3104); and computer (Physical Acoustics Corp. model 3000). The transducer was coupled and bonded to the 4340 steel plate using RTV silicone rubber (General Electric Co.).

The acoustic emission was monitored continuously during the deposition of HC and LC chromium. The threshold for signal detection was set identically for both plating conditions to insure an accurate comparison. The acoustic emission was also monitored during a control experiment in which hydrogen was evolved at the steel surface in a solution of 250 g/l  $\text{CrO}_3$  at a current density of 30 A/dm<sup>2</sup>. Visible bubbling could be observed at the steel surface. Since very little chromium was deposited without the sulfuric acid catalyst, the observed signal could be attributed to the evolution of hydrogen.

## RESULTS

Figures 2 and 3 show the emission count rate and cumulative counts as a function of time during the deposition of HC chromium. Note that the count rate is low for the first ten minutes, but increases dramatically thereafter. Another significant feature of the emission during HC plating is the apparent periodicity of spikes in the count rate, as can be seen in Figure 2. The time between spikes is on the order of one minute, corresponding to a change in deposit thickness of about 0.5  $\mu\text{m}$ . One may conclude from this that sudden bursts of cracking activity occur at somewhat regular intervals to relieve stress as the deposit thickens.

The amplitude distribution during the first ten minutes of HC plating is shown in Figure 4, with the number of events in each 0.75 dB interval plotted logarithmically. Strictly speaking, this is not a true amplitude



distribution because the signals have been sorted into ranges of constant width on the dB scale rather than on a voltage scale. Nevertheless, the type of distribution shown is commonly used (ref 4) and will be used throughout this report.

No events are shown below 60 dB because this represents the threshold cutoff. The distribution dropped off almost completely by about 70 dB. As the deposition process continued, the shape of the amplitude distribution changed to that shown in Figure 5 for the last ten minutes of the one hour HC plating experiment. The distribution in this case dropped off with a much lower slope and included significantly more higher amplitude signals.

For comparison, the amplitude distribution during the hydrogen evolution experiment is shown in Figure 6. Note the striking similarity to Figure 4, the distribution during the first ten minutes of HC plating.

A question arises as to what might be happening during the first few minutes of the HC plating process. Because of the low count rate and the similarity to the first ten minutes' amplitude distribution for hydrogen evolution, one might erroneously conclude that no cracking occurs during this period. To gain insight into this period of plating, the HC deposition process was halted after eight minutes and the deposit examined for cracks. As Figure 7 shows, the deposit is about 1  $\mu\text{m}$  thick and has begun to crack. Therefore, the acoustic emission during this period cannot be attributed entirely to hydrogen evolution.

It is widely accepted that cracking begins when about 0.5  $\mu\text{m}$  of chromium has been deposited, as observed by Takano and Ono, also using acoustic

<sup>4</sup>J. Holt, D. J. Goddard, and I. G. Palmer, "Methods of Measurement and Assessment of the Acoustic Emission Activity From the Deformation of Low Alloy Steels," NDT International, Vol. 14, No. 2, April 1981, pp. 49-58.

emission (ref 6). Because of the possibility of emission due to hydrogen evolution during the early minutes of deposition, our data is not able to determine precisely when cracking actually begins.

Figure 3 shows the cumulative counts as a function of time during the deposition of LC chromium. The count rate is quite low, compared to that for HC chromium, during the duration of the deposition process. The amplitude distribution during the entire one hour LC plating experiment is shown in Figure 8. The shape of the distribution is similar to that for hydrogen evolution, Figure 6. One may safely conclude that the acoustic emissions during the deposition of LC chromium are due to the evolution of hydrogen rather than cracking in the deposit.

The plating of the intermediately cracked chromium yielded a cumulative counts versus time curve as shown in Figure 3. As expected, the count rate lies between that for HC and that for LC.

Figure 9 shows typical photomicrographs which illustrate the degree of cracking occurring in HC chromium and the deposit of intermediate crack density as well as the absence of cracks in LC chromium. No further effort was made to quantify the actual crack densities.

## CONCLUSIONS

On the basis of our observations, one may conclude that the electrodeposition of HC chromium leads to a substantially different acoustic emission signature than the electrodeposition of LC chromium. The clearest difference

<sup>6</sup>O. Takano and K. Ono, "Acoustic Emission During Electro and Electroless Plating," Technical Report No. UCLA-ENG-7473, California University, Los Angeles School of Engineering and Applied Science, Los Angeles, CA, July 1974.

is in the count rates, where the cracking occurring in the HC deposit leads to a comparatively high count rate.

Regarding the cracking process, one may conclude from our data that after the first few minutes of deposition, sudden bursts of cracking occur at somewhat regular intervals to relieve stress as the deposit thickens. Those intervals correspond to a change in thickness of about  $0.5 \mu\text{m}$ .

It is widely accepted that cracks in HC chromium develop only after an initial deposit thickness of  $0.5 \mu\text{m}$  is exceeded. Because of the possibility of emissions due to hydrogen evolution, we were unable to determine such a critical thickness precisely. Further work, involving frequency analyses of the signals, may show more precisely when cracking actually begins.

Regarding the use of acoustic emission, one may conclude that the technique is of value in directly monitoring cracking during the deposition process. Without such a technique, an experimenter would remain ignorant of what might be taking place in the deposit until it is physically removed from the electrolyte for examination. Upon further study, it may even be possible to determine the size and distribution of cracks using the emission amplitudes and emission rate (ref 7), though such quantitative information may prove difficult to obtain.

The difficulty with which acoustic emission yields precise quantitative information about cracking is one of its limitations. Those who use acoustic emission are prone to accept this limitation and appear to make successful use of semi-quantitative information.

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<sup>7</sup>C. T. Peters and C. Larson, "Acoustic Emission to Monitor Electrodeposition," NDT International, Vol. 9, No. 4, August 1976.

## SUMMARY

In this study, we have used acoustic emission to study crack formation during the electrodeposition of chromium under conditions which lead to the deposition of HC and of LC chromium. Several characteristics of the acoustic differences were observed between the emissions from HC chromium and those from LC chromium. The acoustic emission technique detected the onset of cracking and yielded information regarding the extent of cracking. Such information, especially when acquired during the actual deposition process, can be an invaluable aid in determining the quality of the deposit and in developing improved chromium coatings.

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1. A. G. Beattie, "Acoustic Emission, Principles and Instrumentation," Journal of Acoustic Emission, Vol. 2, April 1983, pp. 95-128.
2. B. J. Brindley, J. Holt, and I. G. Palmer, "Acoustic Emission III - The Use of Ring-Down Counting," Non-Destructive Testing, Vol. 6, No. 6, December 1973.
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6. O. Takano and K. Ono, "Acoustic Emission During Electro and Electroless Plating," Technical Report No. UCLA-ENG-7473, California University, Los Angeles School of Engineering and Applied Science, Los Angeles, CA, July 1974.
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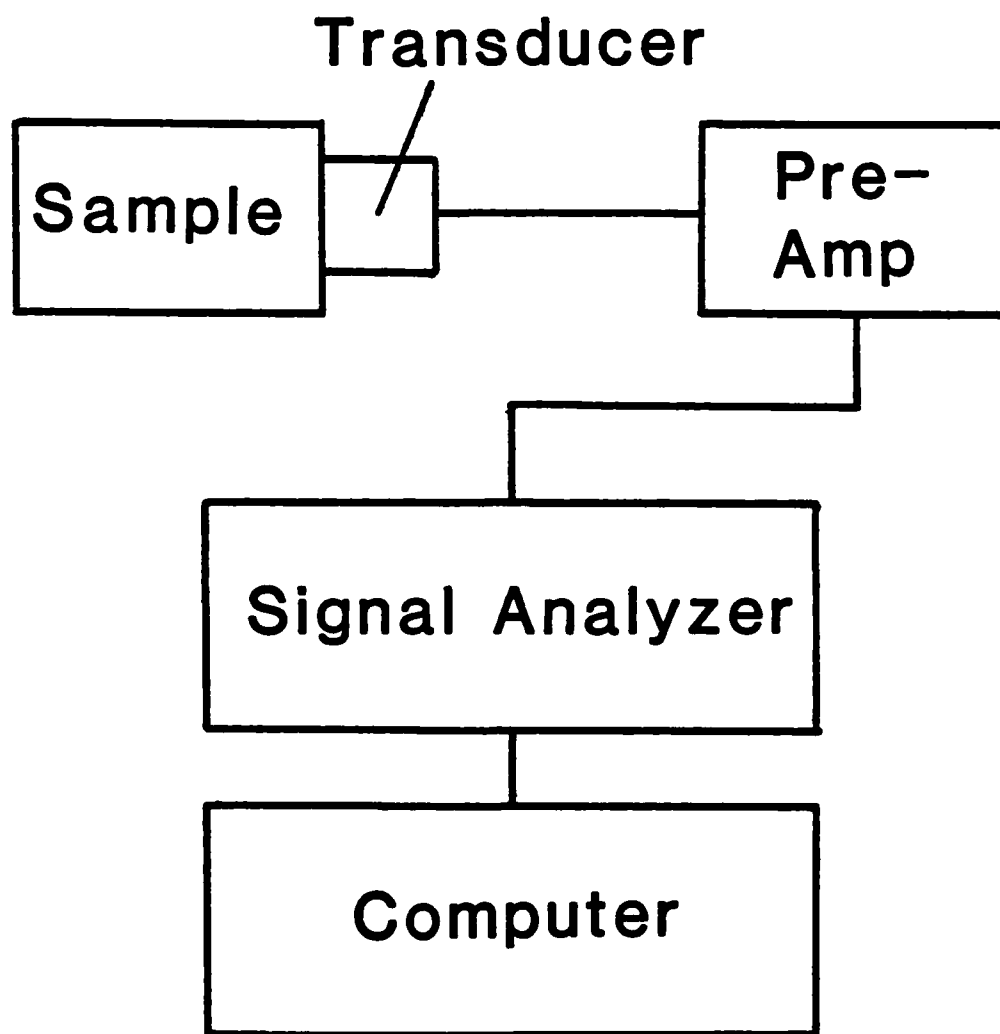


Figure 1. Schematic diagram of the experimental apparatus.

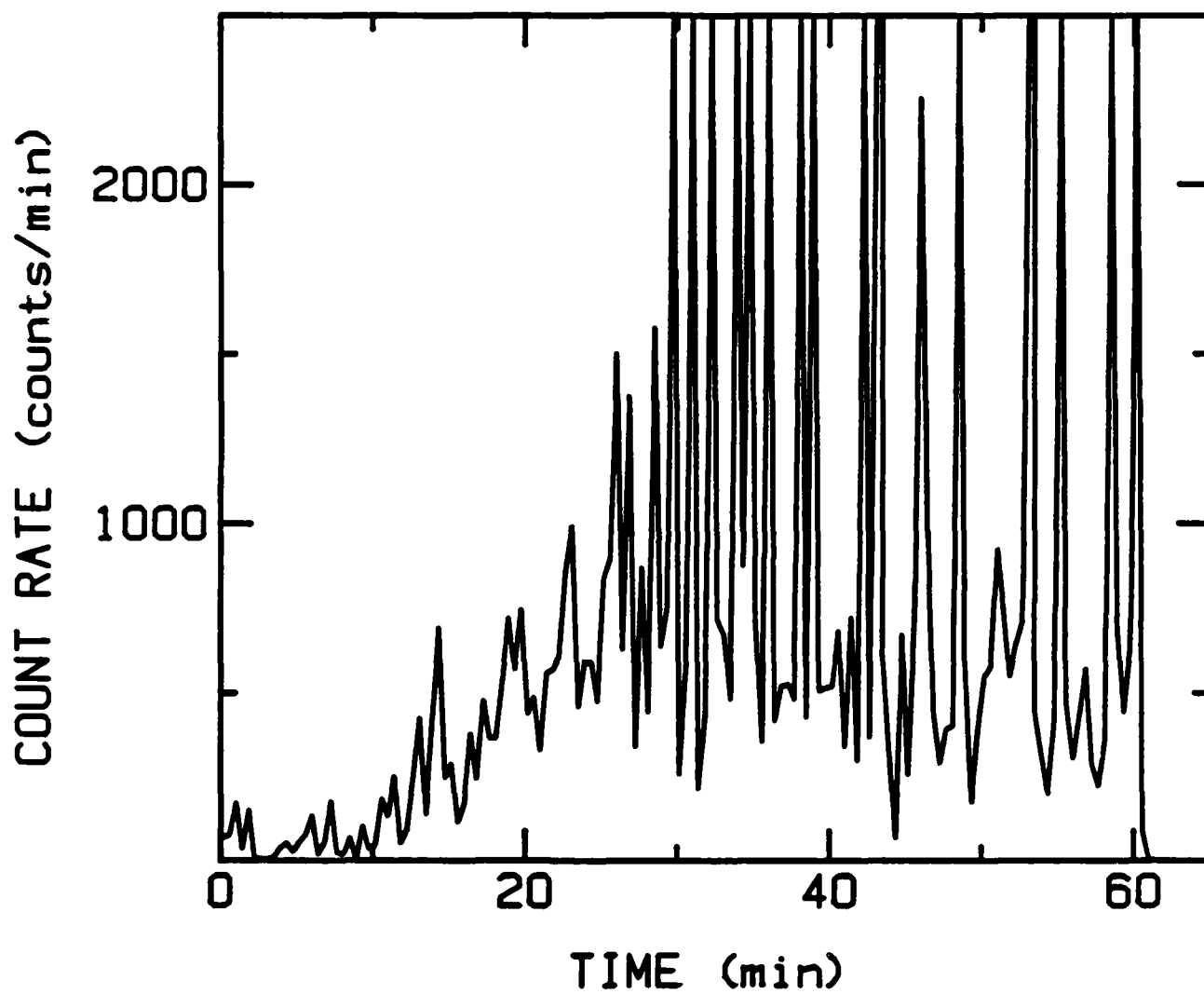


Figure 2. Count rate versus time during the deposition of HC, LC, and intermediately cracked chromium.

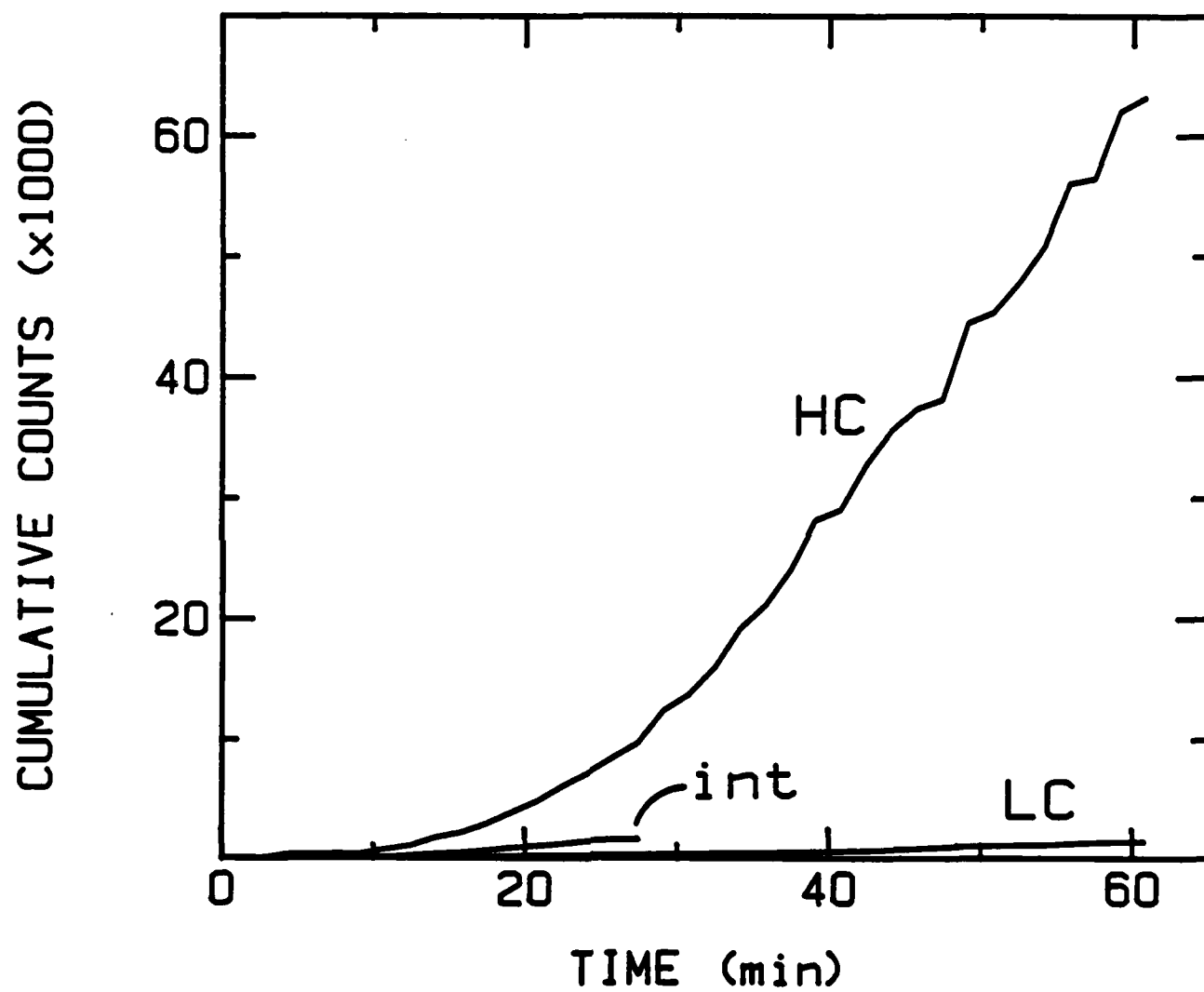


Figure 3. Cumulative counts versus time during the deposition of HC chromium.



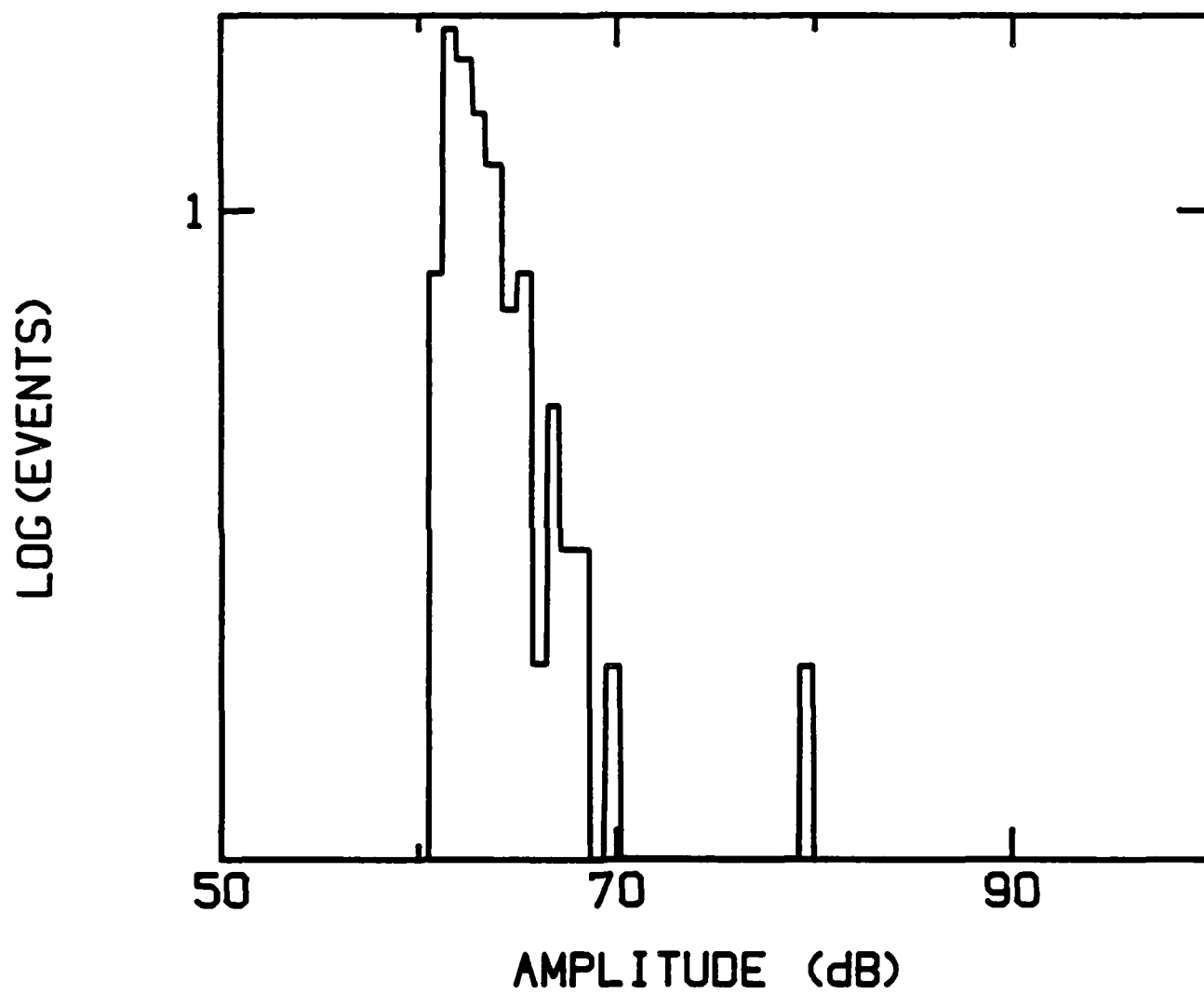


Figure 4. Acoustic emission amplitude distribution during the first ten minutes of HC chromium deposition process.

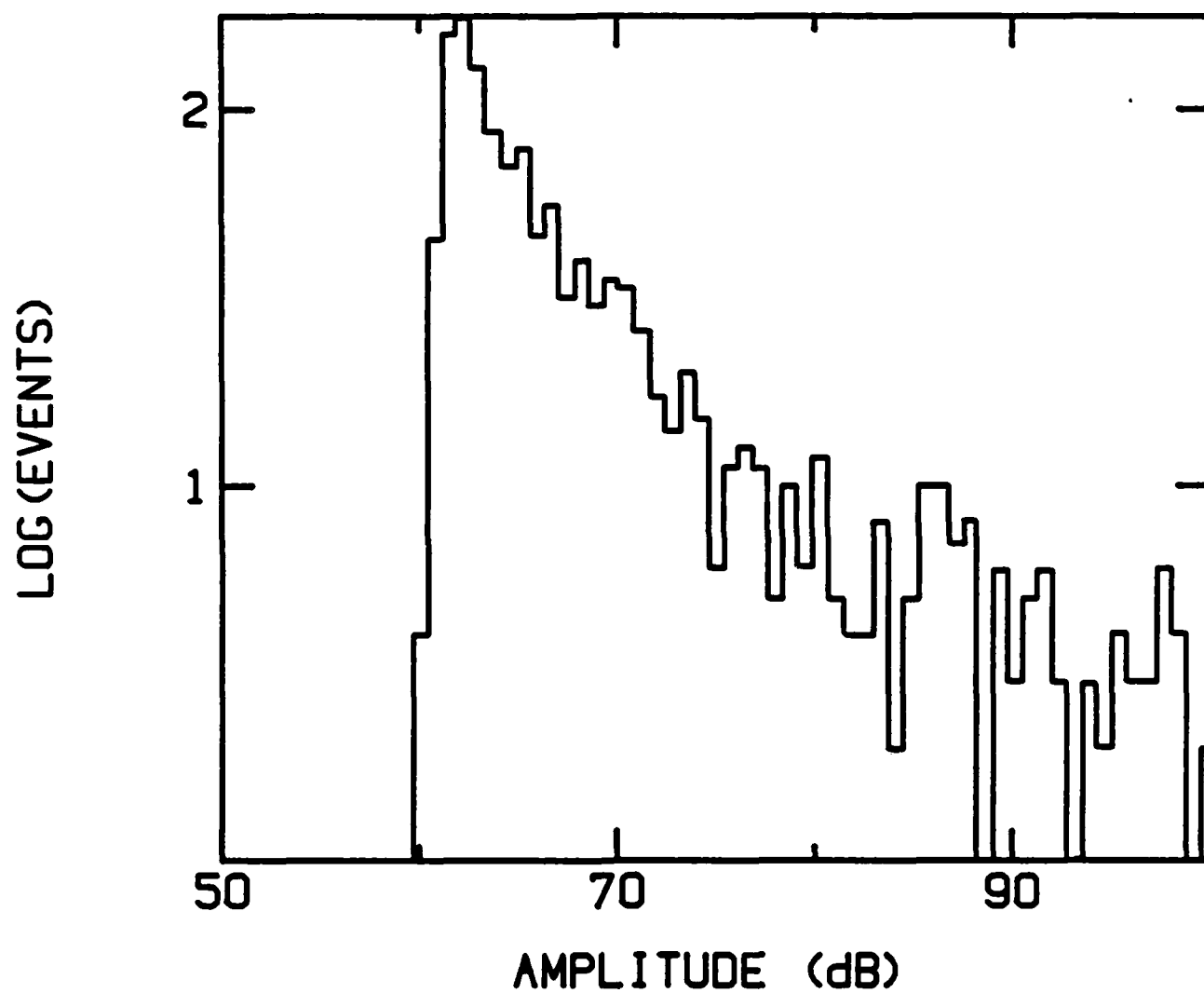


Figure 5. Acoustic emission amplitude distribution between 50 and 60 minutes after beginning the HC chromium deposition process.

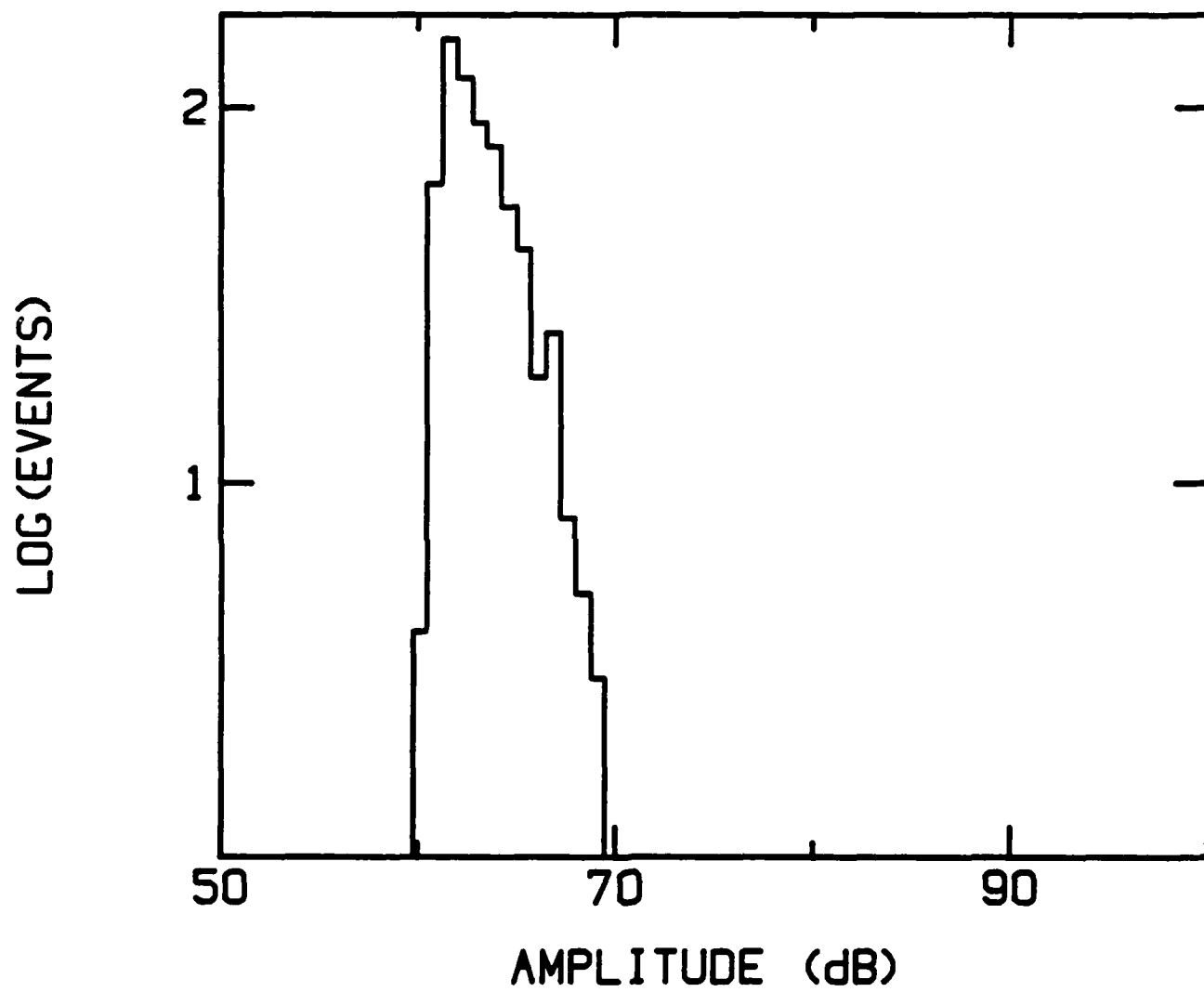


Figure 6. Acoustic emission amplitude distribution during hydrogen evolution.

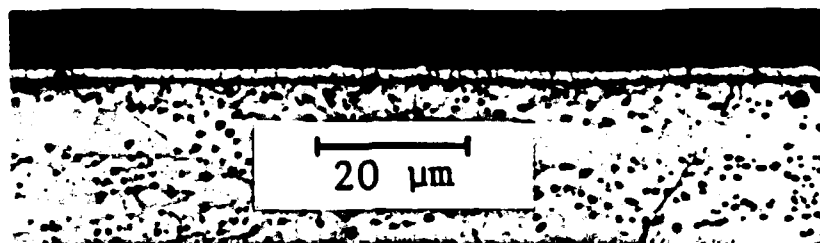


Figure 7. Photomicrograph of HC chromium after first eight minutes of plating.

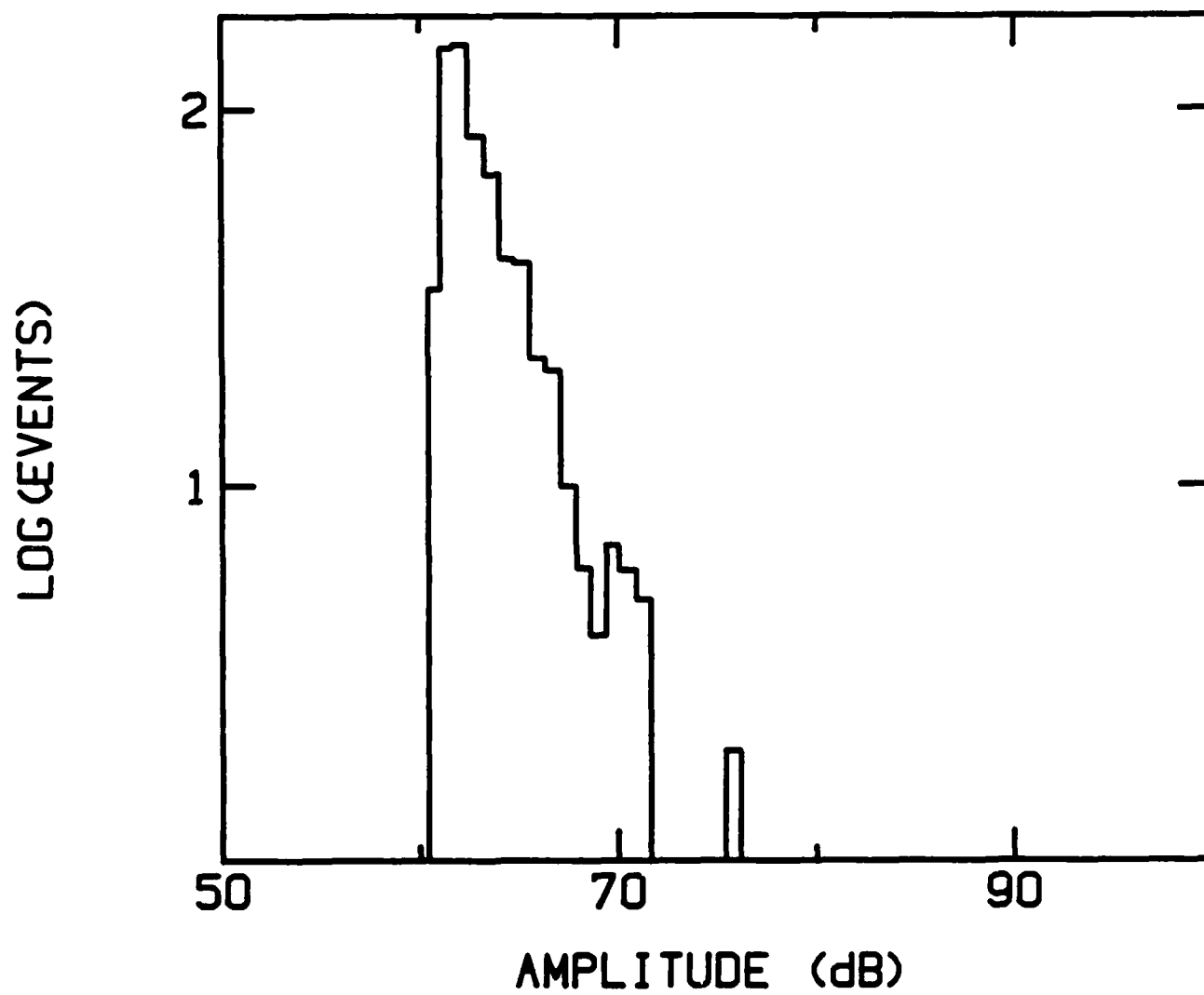


Figure 8. Acoustic emission amplitude distribution during the deposition of LC chromium.

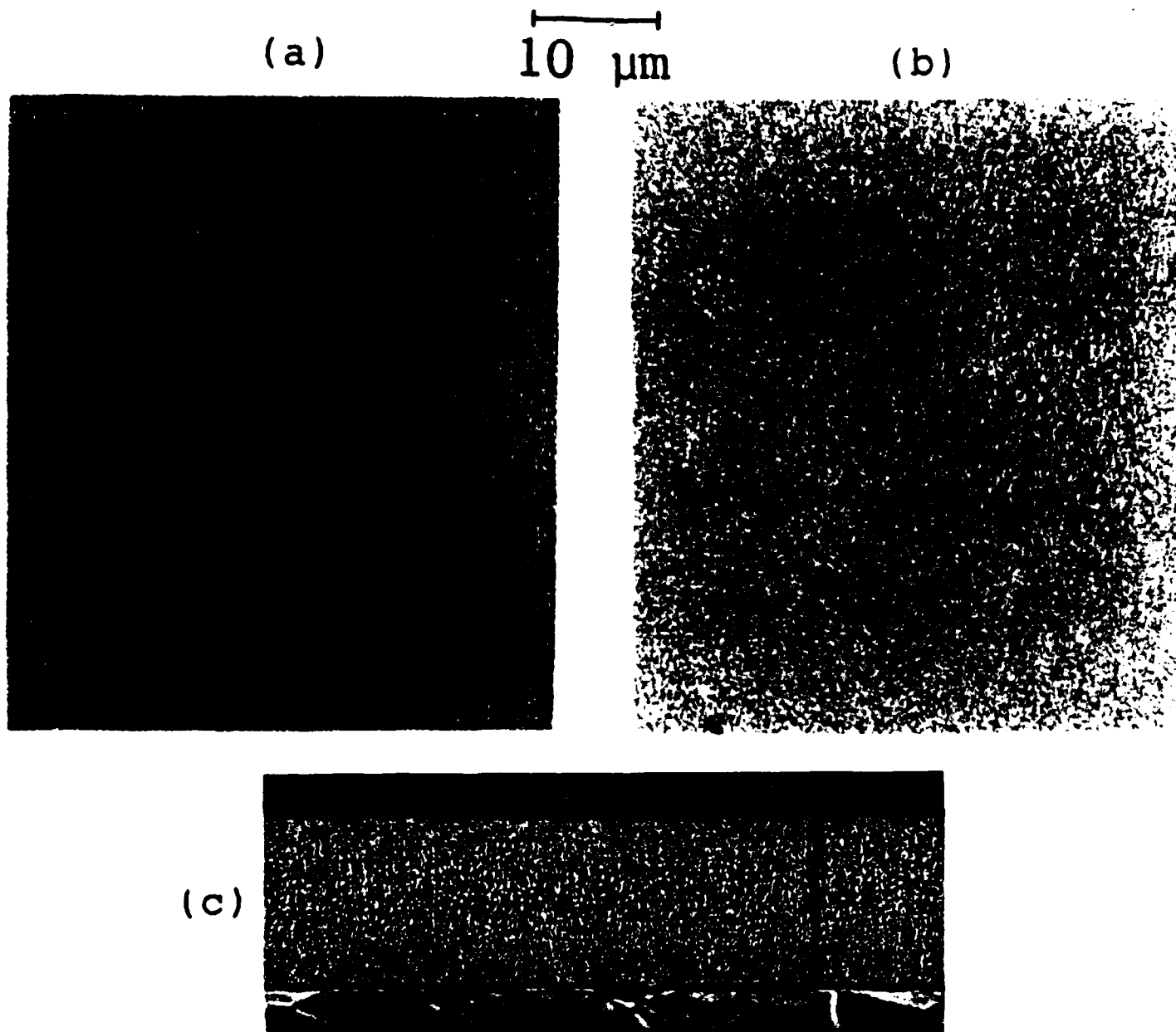


Figure 9. Typical photomicrographs of HC chromium, LC chromium, and the intermediately cracked chromium.

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